



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Scott R. Conley, et al

ELECTROLUMINESCENT DEVICE
WITH ANTHRACENE DERIVATIVE
HOST

Serial No. 10/809,064

Filed 25 March 2004

Commissioner for Patents
P.O. Box 1450
Alexandria, VA. 22313-1450

Sir:

Group Art Unit: 1712

Examiner: Timothy J. Kugel

I hereby certify that this correspondence is being
deposited today with the United States Postal
Service as first class mail in an envelope addressed
to Commissioner For Patents, P.O. Box 1450,
Alexandria, VA 22313-1450.

Deidra L. Mack
Deidra L. Mack

April 21, 2006
Date

DECLARATION UNDER RULE 131

The undersigned, Scott R. Conley, declares that:

He is a co-inventor in the present application.

He is now and has been, since the date of the present invention, an
employee of the Eastman Kodak Company.

In accordance with Kodak's established procedure for documenting
inventions, notebook entries were made by me and my laboratory assistant
documenting the preparation of anthracene derivative compounds to be tested as
hosts for OLED devices. These numbered pages are dated prior to September
2003 though the actual dates have been excised and are enclosed as Exhibit A. I
have added an identification of the corresponding compound of the application
within a red circle on the photocopy to assist in verifying the identity of the
compounds made. The synthesis efforts in these pages extend over a period of
about six weeks.

Further, in accordance with Kodak's routine for preparing and testing
OLED materials, the described chemical host materials were submitted to the
group responsible for sample preparations (multilayer chemical vapor deposition)
and samples were prepared and tested as shown in Exhibit B. These test were

carried out diligently within three months of completion of the chemical syntheses. the test results correspond to examples in the application.

It is believed that the foregoing establishes the inventive concept prior to September 2003 and a diligent reduction to practice thereafter.

The undersigned declares further that all statements made herein of the undersigned's own knowledge are true and all statements made on information and belief are believed to be true. These statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.


Scott R. Conley

Date: April 20, 2006

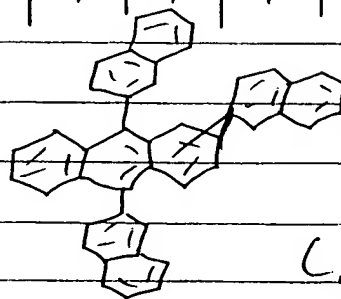
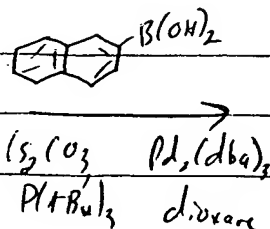
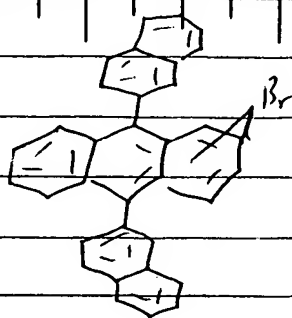
EASTMAN KODAK COMPANY

Notebook No. _____

Date _____

Exhibit A

Problem: _____



Inv-3

 $C_{44}H_{29}$

	MW	eq	m.mole	g
Br-ADN	509.44	1	0.79	0.4
boronic acid	171.99	1.5	1.18	0.2
$Pd_2(dba)_3$	915.70	0.15	0.12	0.1
$(C_5H_5O_2)$	325.82	0.62	1.57	0.51
$Pt(Bu)_3$	202.32	0.36	0.28	0.06 $d^{20.812}$
$Pt(dba)_3$				0.07mL

solvent

dioxane 1.6mL \rightarrow 0.5M

Yield

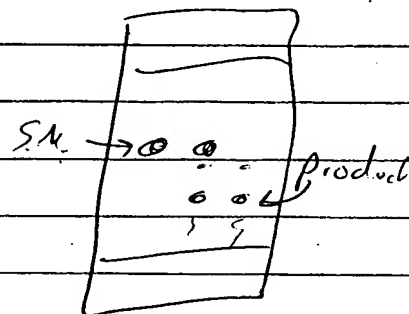
Product	557.71	1	0.79	0.44
---------	--------	---	------	------

Procedure

Stir Br-ADN (0.4g), boronic acid (0.2g), $Pd_2(dba)_3$ (0.1g), $(C_5H_5O_2)$ (0.5g), and $Pt(Bu)_3$ (0.07mL) in dioxane (1.6mL). Heat to 50°C overnight. Extract w/ CH_2Cl_2 . Dry w/ $MgSO_4$. Filter conc.

column chromatography (20% CH_2Cl_2 / 80% heptane)

~ 0.08g.



Signature

Scott Carls

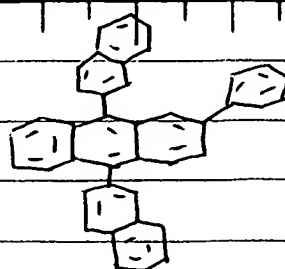
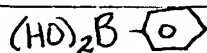
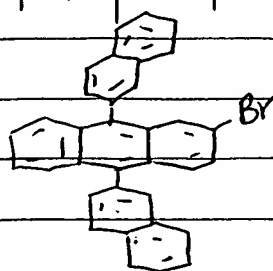
The foregoing disclosed to me on _____

Witness

J. H. C.

BEST AVAILABLE COPY

Problem: _____

C₄₀H₂₆O

Inv-5

	MW	eq	mmol	g
Bromo-ADN	509.44	1	3.9	2
Phenylboronic acid	121.93	1	3.9	0.48
Pd ₂ (dba) ₃	915.70	.015	0.06	0.05
P(tBu) ₃	262.32	.036	0.14	0.03 d=0.82 0.03 mL
Cs ₂ CO ₃	325.82	2	7.8	2.54
Solvent	Dioxane 0.1M 40mL			
Yield				
Product	506.46	1	3.9	1.9

- Combine all in 100mL dried RB w/ N₂. Heat to 80°C Stir overnight.
- Cool to RT. Add H₂O : extract w/ CH₂Cl₂. Dry w/ MgSO₄, filter, conc.
- Column 5% CH₂Cl₂ / 95% heptane. 4 total columns.
- Can see in MS C0083-163-2, NMR OK C0083-163-2, 26 H⁺,
- HPLC 92% purity. C0083-163-2
- Sublime @ 190°C. Give to Mary Store 20mg.

KP 15226-6/00

Signature

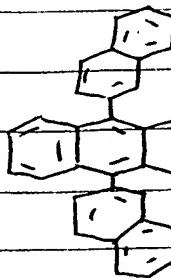
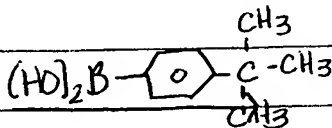
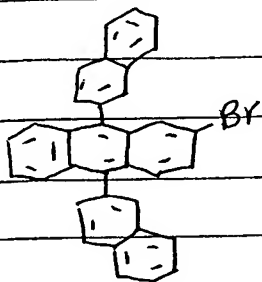
Andrea Scudell

The foregoing disclosed to me on _____

Witness

BEST AVAILABLE COPY

Problem:

C₄₄H₃₄

Inv-7

	MW	eq	mmol	g
Bromo-ADN	509.44	1	5.9	3
4-t-butylphenyl acid	178.0	1	5.9	1.1
Pd ₂ (dba) ₃	915.70	0.15	0.09	0.08
P(tBu) ₃	202.32	0.36	0.2	0.04 d=0.82 0.05 mL
Cs ₂ CO ₃	325.82	2	11.8	3.8
Solvent	Dioxane 0.14 → 59 mL			
Yield				0.82 25%
Product	562.75	1	5.9	3.3

- Combine all in 250 mL heated RB/stir. Heat to 80°C w/ N₂. Stir overnight.

- Cool to RT. Add H₂O, extract w/ CH₂Cl₂. Column 5% CH₂Cl₂/95% heptane.

- 94% pure Matl. by HPLC. CC0083-1165-1.

- MS CC0083-1165-1, Major product.

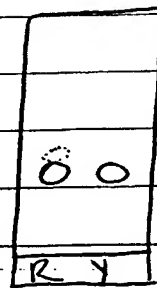
- Sublime (B) → 245°C

2 bands → red : yellow.

Red: 0.18g, slight impurity above product spot.

red/yellow: 0.41g, yellow is more pure than red.

Store. Run rxn. again. HPLC 1165R=90%, HPLC 1165Y=95%



ADN HPLC=98%

KP 15226-6/00

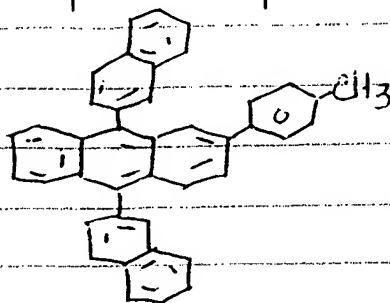
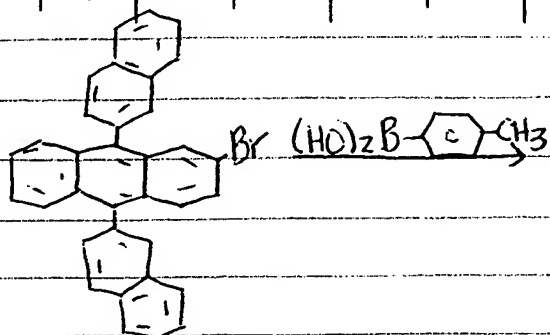
Signature

Andrew Seidell

The foregoing disclosed to me on

Witness

BEST AVAILABLE COPY

C₄₁H₂₈

Inv-1

	MW	eq	mmol	g
Bromo-ADN	509.44	1	5.9	3
p-tolylboronic acid	135.96	1	5.9	0.80
Pd ₂ (dba) ₃	915.70	.015	0.09	0.08
P(t-Bu) ₃	202.32	.036	0.2	0.04d=0.82 0.05mL
Cs ₂ CO ₃	325.82	2	11.8	3.8
Solvent	Dioxane 0.1M → 59mL			
Yield				0.43 14%
Product	520.67	1	5.9	307

- Combine all in 250mL. Heat to 80°C, stir w/ N₂ overnight.
- Stir for 3 days. Add H₂O, extract w/ CH₂Cl₂. Dry w/ MgSO₄, filter, conc, dry. Very bromine in color.
- Column 95% heptane / 5% EtOAc, yellow/red in color.
- Mass spec C00083-1168 confirms mass, also contains mass of boronic acid.
- LC C00083-1168 93% pure. Looks most pure of all crops.
- 1.06g SM remaining, 0.43 product. Sublime: Begin at 190°C, end at 210°C.
- Mass spec C00083-1168 P

INMR, LC C00083-1168 AS after sublimation

KP 15226-6/00

Signature

Andrea Scuderi

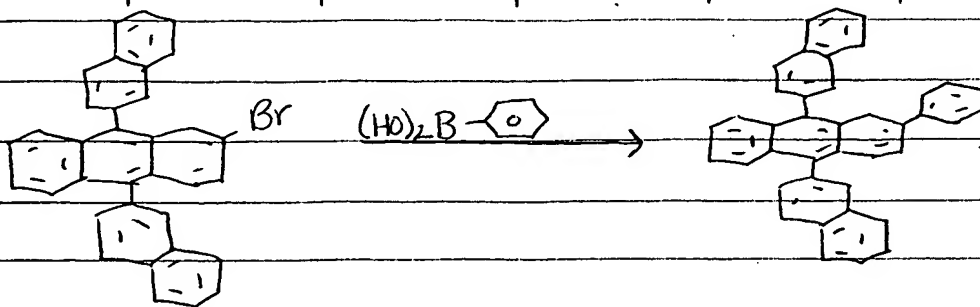
The foregoing disclosed to me on _____

Witness

J. B. T.

BEST AVAILABLE COPY

Problem: _____



~~XXXXXXXXXX~~
 Inv-5

	MW	eq	mmol	g
Bromo-ADN	509.44	1	13.7	7
Phenylboronic Acid	121.93	1	13.7	1.67
$\text{Pd}(\text{dba})_3$	915.70	.015	0.21	0.19
$\text{P}(\text{tBu})_3$	202.32	0.036	0.49	0.099 d=0.82 .12 mL
Cs_2CO_3	325.82	2	27.4	8.9
Solvent	Dioxane 0.1M \rightarrow 137 mL			
Yield				1.44 (after column)
Product	506.46	1	13.7	6.9 21%

- In dried 500 mL RB \rightarrow add all (-Pd). React 10 min, add Pd. Heat to 80°C , heat: stir overnight.

- Add 0.5 equiv. acid to speed up rxn. after it can overnight.

- TLC: progress much faster w/ 1.5 equiv. of acid.

- Cool to RT. Add H_2O , extract w/ CH_2Cl_2 , dry w/ MgSO_4 , filter conc. dry. TLC:

- Column 5% CH_2Cl_2 / 95% heptane. Obtain 1.44 g "pure" matl. by

HPLC. Give to Craig Swanson for Br analysis, 850 ppm Br.

(Is still orange matl.) *hydrogenate to remove Br (Pd/C in DMF) (start w/ 1 g)

- Filter through celite, rinse w/ DMF: CH_2Cl_2 Column to receive. 91 g

yellow matl. Submit to Craig for Br analysis. Store final sublimed matl.

(Still contains red)

di-BrSH
BrSH
ADN
PR

KP 15226-6/00

Signature

Andrea Scudell

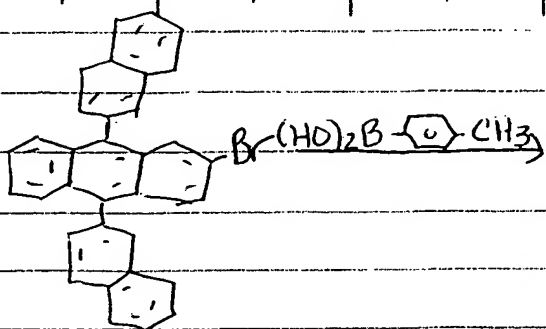
The foregoing disclosed to me on

BEST AVAILABLE COPY

J. J. L.

Date

Problem:



Inv-1

	MW	eq	mmol	g
Bromo-ADN	509.44	1	13.7	7
p-tolylboronic acid	135.96	1	13.7	1.87
Pd ₂ (dba) ₃	915.70	.015	0.21	0.19
P(t-Bu) ₃	202.32	.036	0.49	0.099 d=0.82 0.12ml
Cs ₂ CO ₃	325.82	2	27.4	8.9
Solvent	Dioxane 0.1M → 137ml			
Yield				
Product	520.67	1	13.7	7.1

- Indried 500ml DB. add all -Pd. React 10min. Heat to Reflux overnight. (after adding Pd)
- Cool to RT. Add H₂O, extract w/CH₂Cl₂. Dry w/MgSO₄, filter, conc, dry.
- Column 5% CH₂Cl₂/95% heptane.
- Hydrogenate (start w/ 1.03g) Pd/C in DMF. Overnight.
- Filter through celite. Wash celite w/ CH₂Cl₂, Concentrate.
- Column 5% CH₂Cl₂/95% heptane.

- Craig Swanson for Br analysis. (9/10/03)
- Sublime on 10.23.03

KP 15226-6/00

Signature

Andrea Kodell

Witness

JL

The foregoing disclosed to me on

BEST AVAILABLE COPY

Inu-1

OLED run#:
Completed date:
Operator NB ref:
Run request date:
Originator:
Originator NB ref:
Expermt Objective:

Scott Conley

Test new blue host

Subl. Temp.:

Cell Label (A-F)	A	B	C	D	E	F
Substrate	Sanyo metal coated cell					
Anode	ITO					
Pretreatment	CFx	CFx	CFx	CFx	CFx	CFx
HTL material	NPB					
Thickness (A)	750	750	750	750	750	750
Rate (A/s)	1	1	1	1	1	1
Emitter host						
EMD dopant						
Dopant Volume %	0.75%	0.75%	1.5%	1.5%	3.0%	0.75%
Thickness (A)	1.5	3	3	6	6	1.5
Rate (A/s)						
Rate high/low						
ETL	Alq					
Thickness (A)	350	350	350	350	350	350
Rate (A/s)	/					
Cathode Mg/Ag						
Mg thickness (A)	2000	2000	2000	2000	2000	2000
Mg rate (A)	/	10	10	10	10	10
Ag thickness (A)	200	200	200	200	200	200
Ag rate (A)	/	1	1	1	1	1
Device Area @ 20 m						
Voltage	6.9	7.3	6.5	7.3	6.9	7.5
W/A	0.028	0.027	0.029	0.026	0.026	0.054
Cd/A	2.39	2.49	2.42	2.35	2.22	2.35
CIEx	0.213	0.232	0.211	0.230	0.217	0.146
CIEy	0.289	0.329	0.289	0.326	0.304	0.136
L (cd/m^2)	477.4	498.6	484.8	470.8	443.6	470.6
peak wavelength	452	456	456	456	456	452
Thickness (A)						
PEDOT thickness						
Turnon field						
% drop @ 100 h						
T _{1/2} (Hour)	681	629	1115	772	799	739

BEST AVAILABLE COPY

OLED run#:
 Completed date:
 Operator NB ref:
 Run request date:
 Originator: Scott Conley
 Originator NB ref: CC0525-25
 Experm Objective: Test new blue host

Inv-7

Subl. Temp.: 235 °C

Cell label (A/B)	A	B	C	D	E	F
Substrate	Sanyo metal coated cell					
Anode	ITO					
Pretreatment	CFx	CFx	CFx	CFx	CFx	CFx
HTL material	NBB					
Thickness (Å)	750	750	750	750	750	750
Rate (Å/s)						
Emitted light						
EMI dopant	TBP	TBP	TBP	TBP	TBP	TBP
Dopant Volume %	1.00%	1.00%	2.0%	2.0%	4.0%	1.50%
Thickness (Å)	2	4	4	8	8	3
Rate (Å/s)						
Rate high/low						
ETL	Alq					
Thickness (Å)	350	350	350	350	350	350
Rate (Å/s)	/					
Cathode	Mg/Ag					
Mg thickness (Å)	2000	2000	2000	2000	2000	2000
Mg rate (Å)	/	10	10	10	10	10
Ag thickness (Å)	200	200	200	200	200	200
Ag rate (Å)	/	1	1	1	1	1
Device area @ 20 m/s						
Voltage	6.8	8.4	6.8	8.2	6.8	6.9
W/A	0.049	0.047	0.048	0.045	0.044	0.046
Cd/A	3.53	3.96	3.42	3.71	3.21	2.49
CIEx	0.168	0.176	0.166	0.171	0.166	0.145
CIey	0.252	0.319	0.250	0.310	0.260	0.181
L (cd/m ²)	706.7	791.6	683.6	741.6	642.3	497.2
peak wavelength	464	496	464	464	464	460
Thickness (Å)						
PEDOT thickness						
Tumon field						
% drop @ 100 h						
T _{1/2} (Hour)	866	784	1202	1165	1355	1053

BEST AVAILABLE COPY

OLED-run#:
Completed date:
Operator NB ref:
Run request date:
Originator:
Originator NB ref:
Expermt Objective

Scott Conley

Test new blue host

Inv-1

Subl. Temp.: 230

Cell label (A-F)	A	B	C	D	E	F
Substrate	Sanyo metal coated cell					
Alode	ITO					
Pretreatment	CFx	CFx	CFx	CFx	CFx	CFx
HTL material	NPB	NPB	NPB	NPB	NPB	NPB
Thickness (A)	750	750	750	750	750	750
Rate (A/s)	1	1	1	1	1	1
Emitter host	TBADN					TBADN
Thickness	100	100	100	100	100	100
Rate (A/s)	1	1	1	1	1	1
EMI Dopant	TBP	TBP	TBP	TBP	TBP	TBP
Dopant Volume %	1.00%	1.00%	2.0%	2.0%	4.0%	1.50%
Thickness (A)	2	4	4	8	8	3
Rate (A/s)	1	1	1	1	1	1
Rate high/low						
ETL	Alq	Alq	Alq	Alq	Alq	Alq
Thickness (A)	350	350	350	350	350	350
Rate (A/s)	1	1	1	1	1	1
Cathode	Mg/Ag	Mg/Ag	Mg/Ag	Mg/Ag	Mg/Ag	Mg/Ag
Mg thickness (A)	2000	2000	2000	2000	2000	2000
Mg rate (A)	10	10	10	10	10	10
Ag thickness (A)	200	200	200	200	200	200
Ag rate (A)	1	1	1	1	1	1
Device data @ 20 m ²	A	B	C	D	E	F
Voltage	6.5	8.1	6.7	8.0	6.5	7.0
W/A	0.047	0.048	0.047	0.045	0.041	0.044
Cd/A	3.70	4.18	3.66	3.89	3.30	2.40
CIEx	0.176	0.184	0.174	0.180	0.177	0.144
CIey	0.283	0.333	0.280	0.328	0.296	0.184
L (cd/m ²)	739.8	836.9	731.6	778.2	660.8	479.8
peak wavelength	464	496	464	468	464	460
Thickness (A)						
PEDOT thickness						
Tumon field						
% drop @ 100 h						
T _{1/2} (Hour)	1124	1087	1941	2319	2233	791

BEST AVAILABLE COPY

OLED run#:
 Completed date:
 Operator NB ref:
 Run request date:
 Originator:
 Originator NB ref:
 Expermt Objective

Scott Conley

Test new blue host

Subl. Temp.: 228

Inv-5

Cell label (A-E)	A	B	C	D	E	F
Substrate	Samsung Materials Coated PET					
Address (FE)						
Pretreatment	CFx	DFx	DFx	DFx	CFx	CFx
HTL material	NISB					
Thickness (A)	250	250	250	250	250	250
Rate (A/s)						
Emitter host	ABADN					
EMI dopant	TBP	TBP	TBP	TBP	TBP	TBP
Dopant Volume %	1.00%	1.00%	2.0%	2.0%	4.0%	1.60%
Thickness (A)	2	4	4	8	8	3
Rate (A/s)						
Rate high/low						
EMI	Ag					
Thickness (A)	350	350	350	350	350	350
Rate (A/s)	/					
Cathode M/A/g						
Mg thickness (A)	2000	2000	2000	2000	2000	2000
Mg rate (A)	/	10	10	10	10	10
Ag thickness (A)	200	200	200	200	200	200
Ag rate (A)	/	1	1	1	1	1
Device data @ 20 mV						
Voltage	7.1	8.7	7.5	8.1	7.0	7.4
W/A	0.049	0.055	0.048	0.052	0.042	0.052
Cd/A	3.43	4.33	3.40	4.06	3.15	2.85
CIEx	0.161	0.163	0.160	0.162	0.165	0.141
CIEy	0.250	0.296	0.254	0.299	0.275	0.191
L (cd/m^2)	686.5	865.1	679.4	811.7	630.8	570.3
peak wavelength	464	468	464	468	464	464
Thickness (A)						
PEDOT thickness						
Tumon field						
% drop @ 100 h						
T ₁₂ (Hour)	5737	3869	5524	14260	6120	2541

BEST AVAILABLE COPY